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## P/ INT COOPERATION TREAT

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner  
 US Department of Commerce  
 United States Patent and Trademark  
 Office, PCT  
 2011 South Clark Place Room  
 CP2/5C24  
 Arlington, VA 22202  
 ETATS-UNIS D'AMERIQUE  
 in its capacity as elected Office

Date of mailing (day/month/year) 14 February 2001 (14.02.01)	
International application No. PCT/EP00/05341	Applicant's or agent's file reference MPD315/PCT/RGMS
International filing date (day/month/year) 09 June 2000 (09.06.00)	Priority date (day/month/year) 10 June 1999 (10.06.99)
Applicant HATCHMAN, Kevan et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:  
 27 December 2000 (27.12.00)

☐ in a notice effecting later election filed with the International Bureau on:  
 \_\_\_\_\_

2. The election ☒ was  
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland  Facsimile No.: (41-22) 740.14.35	Authorized officer  Claudio Borton  Telephone No.: (41-22) 338.83.38
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## PCT COOPERATION TREATY

PCT

## NOTIFICATION RELATING TO PRIORITY CLAIM

(PCT Rules 26bis.1 and 26bis.2 and  
Administrative Instructions, Sections 402 and 409)

From the INTERNATIONAL BUREAU

To:

SAVIDGE, Roger, Gordon, Madgwick  
Rhodia Consumer Specialties Limited  
210-222 Hagley Road West  
Oldbury  
West Midlands B68 0NN  
ROYAUME-UNI

Date of mailing (day/month/year) 18 October 2000 (18.10.00)	
Applicant's or agent's file reference MPD315/PCT/RGMS	<b>IMPORTANT NOTIFICATION</b>
International application No. PCT/EP00/05341	International filing date (day/month/year) 09 June 2000 (09.06.00)
Applicant RHODIA CONSUMER SPECIALTIES LIMITED et al	

The applicant is hereby **notified** of the following in respect of the priority claim(s) made in the international application.

1. ☒ **Correction of priority claim.** In accordance with the applicant's notice received on: 06 October 2000 (06.10.00), the following priority claim has been corrected to read as follows:  
GB 10 June 1999 (10.06.99) 9913408.2  
☐ even though the indication of the number of the earlier application is missing.  
☐ even though the following indication in the priority claim is not the same as the corresponding indication appearing in the priority document:
2. ☐ **Addition of priority claim.** In accordance with the applicant's notice received on: , the following priority claim has been added:  
☐ even though the indication of the number of the earlier application is missing.  
☐ even though the following indication in the priority claim is not the same as the corresponding indication appearing in the priority document:
3. ☐ As a result of the correction and/or addition of (a) priority claim(s) under items 1 and/or 2, the (earliest) priority date is:
4. ☐ **Priority claim considered not to have been made.**  
☐ The applicant failed to respond to the Invitation under Rule 26bis.2(a) (Form PCT/IB/316) within the prescribed time limit.  
☐ The applicant's notice was received after the expiration of the prescribed time limit under Rule 26bis.1(a).  
☐ The applicant's notice failed to correct the priority claim so as to comply with the requirements of Rule 4.10.  
 The applicant may, before the technical preparations for international publication have been completed and subject to the payment of a fee, request the International Bureau to publish, together with the international application, information concerning the priority claim. See Rule 26bis.2(c) and the PCT Applicant's Guide, Volume I, Annex B2(II).
5. ☐ In case where **multiple priorities** have been claimed, the above item(s) relate to the following priority claim(s):
6. A copy of this notification has been sent to the receiving Office and  
☒ to the International Searching Authority (where the international search report has not yet been issued).  
☒ the designated Offices (which have already been notified of the receipt of the record copy).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer  Athina Nickitas-Etienne
Facsimile No. (41-22) 740.14.35	Telephone No. (41-22) 338.83.38

**PCT**

REC 16 OCT 2001

WIPO

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**INTERNATIONAL PRELIMINARY EXAMINATION REPORT**

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference MPD315/PCT/RGMS	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/EP00/05341	International filing date (day/month/year) 09/06/2000	Priority date (day/month/year) 10/06/1999
International Patent Classification (IPC) or national classification and IPC A61K7/00		
Applicant RHODIA CONSUMER SPECIALTIES LIMITED TRADING... et		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 5 sheets, including this cover sheet.

- ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☒ Certain observations on the international application

Date of submission of the demand  27/12/2000	Date of completion of this report  10. 10. 01
Name and mailing address of the international preliminary examining authority:   European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  Pregetter, M  Telephone No. +49 89 2399 8719  

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/EP00/05341

## I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

### Description, pages:

1-25 as originally filed

### Claims, No.:

1-5 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/EP00/05341

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes:	Claims	
	No:	Claims	1-5
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-5
Industrial applicability (IA)	Yes:	Claims	1-5
	No:	Claims	

**2. Citations and explanations  
see separate sheet**

**VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:  
**see separate sheet**

**R It m V**

Reasoned statement under Article 35 (2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Reference is made to the following documents:

D1: DE 14 67 825 A (CHESEBROUGH\_PONDS INC.) 2 January 1969 (1969-01-02)

- 2.1. The subject-matter of present claim 1 is not new according to Article 33(2) PCT. Document D1 already describes compositions comprising water, mineral oil, oil soluble surfactants and hydrophilic surfactants.  
The compositions of D1 are described as transparent mineral oil-water-gels. The main use of these gels is as a "Frisiermittel".  
The subject-matter of claim 1 cannot be clearly delimited from the compositions described in D1. See also the PCT-Guidelines III-4.8.  
Example 1 discloses a composition comprising 20% mineral oil, 15% surfactant with an HLB value of between 12.7 and 15.0, 10% surfactant with an HLB value of between 1.6 and 7.6. The ratio of oil to oil soluble surfactant is 2:1, the ratio of oils soluble to hydrophilic surfactant is 1:1.5.  
It is stated that 60°C lies just above the  $I_1/L_1$  transition temperature.
- 2.2. With regard to dependent claims 2-4 it is noted that a positive opinion can only be given, if dependent claims refer to independent claims that meet the requirements of the PCT.
3. The subject-matter of present claim 5 is not new according to Article 33(2) PCT. Document D1 describes a method for preparing compositions according to present claim 1 wherein mineral oil and the oil soluble surfactant are mixed and then a mixture of water and the hydrophilic surfactant is added. Water is added after the resulting mixture has been heated to a temperature above the "Gelpunkt" which is the  $I_1/L_1$  transition temperature. A cooling step is implicit. (p.10, 3rd paragraph - p.11, 2nd paragraph).

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/EP00/05341

**Re Item VIII**

Certain observations on the international application

1. The subject-matter of present claim 1 is not clear (Article 6 PCT)..
  - 1.1. The expression: "a proportion ..... based on the weight of ..." is not clear. Present claim 1 has been read as defining:
    - a) a proportion of from 8:1 to 1:5 of oil to oil soluble surfactant; and
    - b) a proportion of from 1:1 to 1:30 of oil soluble surfactant to hydrophilic surfactant.This interpretation of claim 1 is based on p.11, 3rd paragraph.
  - 1.2. The statement "adapted to form an  $I_1$  phase having  $I_1/L_1$  transition temperature greater than 25°C is not clear. Either the compounds comprised in the composition result in a composition with the defined effect, or an essential feature defining the claimed composition is missing.  
At present, all compositions comprising the defined compounds in the described proportions are considered to be relevant.



## PATENT COOPERATION TREATY

PCT

From the INTERNATIONAL BUREAU

NOTIFICATION CONCERNING  
SUBMISSION OR TRANSMITTAL  
OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

T :

SAVIDGE, Roger, Gordon, Madgwick  
Rhodia Consumer Specialties Limited  
210-222 Hagley Road West  
Oldbury  
West Midlands B68 0NN  
ROYAUME-UNI

Date of mailing (day/month/year) 18 October 2000 (18.10.00)	<b>IMPORTANT NOTIFICATION</b>
Applicant's or agent's file reference MPD315/PCT/RGMS	
International application No. PCT/EP00/05341	
International publication date (day/month/year) Not yet published	
Applicant RHODIA CONSUMER SPECIALTIES LIMITED et al	

- The applicant is hereby notified of the date of receipt (except where the letters "NR" appear in the right-hand column) by the International Bureau of the priority document(s) relating to the earlier application(s) indicated below. Unless otherwise indicated by an asterisk appearing next to a date of receipt, or by the letters "NR", in the right-hand column, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
- This updates and replaces any previously issued notification concerning submission or transmittal of priority documents.
- An asterisk (\*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b). In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
- The letters "NR" appearing in the right-hand column denote a priority document which was not received by the International Bureau or which the applicant did not request the receiving Office to prepare and transmit to the International Bureau, as provided by Rule 17.1(a) or (b), respectively. In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

<u>Priority date</u>	<u>Priority application No.</u>	<u>Country or regional Office or PCT receiving Office</u>	<u>Date of receipt of priority document</u>
10 June 1999 (10.06.99)	9913408.2	GB	09 Augu 2000 (09.08.00)

The International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland

Authorized officer

Athina Nickitas-Etienne

Facsimile No. (41-22) 740.14.35

Telephone No. (41-22) 338.83.38

## PATENT COOPERATION TREATY

1 Fax copy to J. Zahra 3/1/2001

From the INTERNATIONAL BUREAU

PCT

2 File M. J. Zahra

NOTICE INFORMING THE APPLICANT OF THE  
COMMUNICATION OF THE INTERNATIONAL  
APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

To:

SAVIDGE, Roger, Gordon, Madgwick  
Rhodia Consumer Specialties Limited  
210-222 Hagley Road West  
Oldbury  
West Midlands B68 0NN  
ROYAUME-UNI

Date of mailing (day/month/year)

21 December 2000 (21.12.00)

Applicant's or agent's file reference

MPD315/PCT/BGMS

## IMPORTANT NOTICE

International application No.

PCT/EP00/05341

International filing date (day/month/year)

09 June 2000 (09.06.00)

Priority date (day/month/year)

10 June 1999 (10.06.99)

Applicant

RHODIA CONSUMER SPECIALTIES LIMITED trading as ALBRIGHT & WILSON  
SURFACTANTS EUROPE AND JOHNSON & JO et al

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:

AG, AU, DZ, KP, KR, MZ, US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AE, AL, AM, AP, AT, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EA, EE, EP, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, OA, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW

The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on 21 December 2000 (21.12.00) under No. WO 00/76460

## REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

## REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the national phase, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

The International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland

Facsimile No. (41-22) 740.14.35

Authorized officer

J. Zahra

Telephone No. (41-22) 338.83.38

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
21 December 2000 (21.12.2000)

PCT

(10) International Publication Number  
**WO 00/76460 A2**

- (51) International Patent Classification<sup>7</sup>: **A61K 7/00** **Benjamin** [US/US]; 2028 Farmview Drive, Newton, PA 18940 (US).
- (21) International Application Number: **PCT/EP00/05341**
- (22) International Filing Date: **9 June 2000 (09.06.2000)**
- (25) Filing Language: **English**
- (26) Publication Language: **English**
- (30) Priority Data:  
**9913408.2** **10 June 1999 (10.06.1999)** **GB**
- (71) Applicant (*for all designated States except US*): **RHODIA CONSUMER SPECIALTIES LIMITED** trading as **ALBRIGHT & WILSON SURFACTANTS EUROPE AND JOHNSON & JOHNSON CONSUMER COMPANIES INC** [GB/GB]; 210-222 Hagley Road West, Oldbury, West Midlands B68 0NN (GB).
- (72) Inventors; and
- (75) Inventors/Applicants (*for US only*): **HATCHMAN, Kevan** [GB/GB]; 5 Byland Close, Friarscroft, Bromsgrove, Worcestershire B61 7PL (GB). **LUKENBACH, Elvin** [US/US]; 160 Klinesville Road, Flemington, NJ 08822 (US). **MCCULLOCH, Laura** [GB/US]; 18 Hampton Court 07920, Basking Ridge, NJ (US). **WIEGAND,**
- (74) Agent: **SAVIDGE, Roger, Gordon, Madgwick**; Rhodia Consumer Specialties Limited, 210-222 Hagley Road West, Oldbury, West Midlands B68 0NN (GB).
- (81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.
- (84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
- Published:**  
— *Without international search report and to be republished upon receipt of that report.*
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) Title: **PERSONAL CARE FORMULATIONS**

(57) Abstract: Personal care compositions contain at least 20 % water, 10 to 40 % total surfactant and 2 to 40 % of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (a) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (b) a hydrophilic surfactant having an HLB greater than 11 in a weight proportion of from 1:1 to 1:30 based on the weight of (a), said water surfactant and oil being present in proportions adapted to form an I<sub>1</sub> phase having an I<sub>1</sub>/L<sub>1</sub> transition temperature greater than 25 °C.

WO 00/76460 A2

### **PERSONAL CARE FORMULATIONS**

The present invention relates to shampoo or cleaning compositions suitable for personal care applications in the form of  $I_1$  mesophase systems containing dispersed oil.

Dispersing oil in aqueous shampoo and body wash formulations has presented problems. To prevent the oil phase separating it must either be: (A) emulsified which involves dispersing the oil as colloidal single droplets; (B) microemulsified which involves forming a micellar solution with oil incorporated into surfactant micelles; (C) suspended in a structured surfactant system which typically comprises a dispersion of a surfactant mesophase in aqueous electrolyte; or (D) incorporated into a water soluble solid, pasty or gelatinous composition.

With the exception of microemulsions which are clear, thermodynamically stable, micellar solutions, the foregoing systems are necessarily opaque and contain the oil dispersed in a relatively coarse form, which does not deposit satisfactorily on skin or hair.

However microemulsions are difficult to formulate using the surfactants which are most effective in body wash and other personal care formulations and contain relatively low concentrations of surfactant.

We have now discovered that oil may be stably incorporated into the structure of an  $I_1$  phase to form a clear gel-like composition which contains higher concentrations of surfactant and oil than conventional microemulsions, but which dissolves in water to form a microemulsion. The novel oil-in- $I_1$  compositions also form microemulsions on heating.

Surfactants are known to form mesophases or liquid crystal phases at concentrations above approximately 30% by weight based on the weight of water and surfactant. Mesophases are phases which exhibit a degree of order intermediate between typical liquids and solids. Generally mesophases combine long range order associated with crystals, with fast molecular motion common to liquids.

The formation of detergent mesophases is well documented. Different surfactants and surfactant mixtures differ widely in their ability to form the numerous different mesophases, and in respect of the conditions of concentration and temperature at which they are formed. For a typical surfactant of the type normally used in cleaning products the following mesophases are usually observed. The concentrations given are illustrative only and may vary considerably from one surfactant or surfactant mixture to the next.

Below approximately 30% surfactant an isotropic  $L_1$  phase is formed (with micelles of surfactant in water). Above 30% surfactant many detergents form a M phase which is not normally used in personal care applications since it does not show suitable flow characteristics and is difficult to dissolve or disperse in water. Above the concentrations required to form an M phase, but usually at concentrations of less than 80% active surfactant, i.e. 60%-80% a G-phase is formed. At concentrations higher than those required to form a G-phase, i.e. typically greater than 80% active surfactant, most surfactants form a hydrated solid, and some, especially non-ionic surfactants form a liquid phase containing dispersed micelle sized droplets of water - an inverted micellar solution known as an  $L_2$  phase.  $L_2$  detergent systems do not disperse readily in water and have a tendency to form undesirable gels, e.g. M phases, on dilution.

Some surfactants form viscous isotropic or VI phases. These are immobile phases usually with a vitreous appearance, and have been relatively little studied compared to the other phases discussed above. They have been virtually ignored in the context of formulating cleaning compositions because most of the surfactants and surfactant systems which are commonly used in cleaning compositions do not form VI phases, at least at

normal temperatures, or form them only within narrow concentration ranges and because their known properties as immobile gels has deterred formulators from investigating them. They are recognised as being the most viscous of the lyotropic mesophases.

The different surfactant phases can be recognised by a combination of appearance, rheology, textures under the microscope, electron microscopy and x-ray diffraction or neutron scattering. A detailed description, with illustrations, of the difference textures observable using a polarising microscope, is to be found in the paper by Rosevear JAOCS Vol 31, p628.

The following terms may require explanation or definition:

The "hydrophilic: lipophilic balance", or "HLB" value is used as a measure of the relative affinities of the surfactants for water and oil respectively and correlates with their effectiveness as emulsifiers. HLB value can easily be calculated for alcohol ethoxylates since it is one fifth of the weight percent of ethylene oxide based on the total mole weight. Other surfactants can be assigned equivalent values by applying more complicated formulae or by measuring their relative affinity for water and oil. An HLB value of 20 represents a completely water soluble oil insoluble surfactant, while an HLB value of 0 represents a completely oil soluble and water insoluble surfactant.

"Optically isotropic" surfactant phases do not normally tend to rotate the plane of polarisation of plane polarised light. If a drop of sample is placed between two sheets of optically plane polarising material whose planes are at right angles, and light is shone on to one sheet, optically isotropic surfactant samples do not appear substantially brighter than their surrounding when viewed through the other sheet. Optically anisotropic materials appear substantially brighter. Optically anisotropic mesophases typically show characteristic textures when viewed through a microscope between crossed polarisers, whereas optically isotropic phases usually show a featureless continuum.

"Newtonian liquids" have a viscosity which remains constant at different shear rates. For the purpose of this specification, liquids are considered Newtonian if the viscosity does not vary substantially at shear rates up to  $1000 \text{ sec}^{-1}$ .

"Lamellar" phases are phases which comprise a plurality of bilayers of surfactant arranged in parallel and separated by liquid medium. They include both solid phases and the typical form of the liquid crystal G-phase. G-phases are typically pourable, non-Newtonian, anisotropic products. They are typically viscous-looking, opalescent materials with a characteristic "smeary" appearance on flowing. They form characteristic texture under the polarising microscope and freeze fractured samples have a lamellar appearance under the electron microscope. X-ray diffraction or neutron scattering similarly reveal a lamellar structure, with a principal peak typically between 4 and 10nm, usually 5 to 6nm. Higher order peaks, when present occur at double or higher integral multiples of the Q value of the principal peak. Q is the momentum transfer vector and is related, in the case of lamellar phases, to the repeat spacing d by the equation  $Q = \frac{2n}{d} [\pi]$  where n is the order of the peak.

G-phases, however, can exist in several different forms, including domains of parallel sheets which constitute the bulk of the typical G-phases described above and spherulites formed from a number of concentric spheroidal shells, each of which is a bilayer of surfactant. In this specification the term "lamellar" will be reserved for compositions which are at least partly of the former type. Opaque compositions at least predominantly of the latter type in which the continuous phase is a substantially isotropic solution containing dispersed spherulites are referred to herein as "G-phase compositions". G-phases are sometimes referred to in the literature as  $L_{(\alpha)}$  phases.

$L_1$ -phases are mobile, optically isotropic, and typically Newtonian liquids which show no texture under the polarising microscope. Electron microscopy is capable of resolving the texture of such phases only at very high magnifications, and X-ray or neutron scattering normally gives only a single broad peak typical of a liquid structure, at very small angles

close to the reference beam. The viscosity of an  $L_1$ -phase is usually low, but may rise significantly as the concentration approaches the upper phase boundary.

"M-phases" are typically immobile, anisotropic products resembling low melting point waxes. They give characteristic textures under the polarising microscope, and a hexagonal diffraction pattern by X-ray or neutron diffraction which comprises a major peak, usually at values corresponding to a repeat spacing between 4 and 10nm, and sometimes higher order peaks, the first at a Q-value which is  $3^{0.5}$  times the Q-value of the principal peak and the next double the Q-value of the principal peak. M-phases are sometimes referred to in the literature as H-phases.

The viscous isotropic or "VI" phases are typically immobile, non-Newtonian, optically isotropic and are typically transparent, at least when pure. VI phases have a cubic symmetrical diffraction pattern, under X-ray diffraction or neutron scattering, with a principal peak and higher order peaks at  $2^{0.5}$  and  $3^{0.5}$  times the Q-value of the principal peak.

These cubic liquid crystalline phases are sometimes observed immediately following the micellar phase at ambient temperature as the concentration of surfactant is increased. It has been proposed that such VI phases, sometimes referred to as  $I_1$  phase, may arise from the packing of micelles (probably spherical) in a cubic lattice. At ambient temperature a further increase in surfactant concentration usually results in hexagonal phase ( $M_1$ ), which may be followed by a lamellar phase (G).  $I_1$  phases, when they occur, are usually only observed over a narrow range of concentrations, typically just above those at which the  $L_1$ -phase is formed. The location of such VI phases in a phase diagram suggests that the phase is built up of small closed surfactant aggregates in a water continuum.

An inverse form of the  $I_1$  phase (the  $I_2$  phase) has also been reported, possibly between the inverse hexagonal ( $M_2$ ) and  $L_2$  phases. It consists of a surfactant continuum containing a cubic array of inverted micelles. An alternative form of the VI phase called



the  $V_1$  phase has been observed at concentrations between the M and G phases and may comprise a bicontinuous system. This may exhibit an even higher viscosity than the  $I_1$ . An inverse phase, the  $V_2$  phase, between the G and  $M_2$  phases has also been postulated.

VI phases are typically examples of "ringing gels". When a jar or beaker containing such a phase is sharply struck, a distinctive vibration can be felt in the composition.

The  $I_1/L_1$  transition temperature will be referred to herein as the melting point of the  $I_1$  phase for convenience, although it is not strictly speaking the melting point since the VI phases are not solids.

All references herein to the formation or existence of specific phases or structures are to be construed, unless the context requires otherwise, as references to their formation or existence at 20°C.

Hexagonal gels (M-phase) have been referred to in the prior art as cleaning compositions, e.g. GB 2 179 055, EP I 153 837 and colloidal gels formed with gelling agents such as synthetic polymers or gelatin have also been suggested, e.g. US 4 465 663.

However these compositions cannot be readily dissolved in water to form microemulsions. They are moreover usually opaque and of an unattractive appearance and often require the presence of solvents such as glycols which add to the cost and are environmentally undesirable.

The use of a type of ringing gel to suspend oil for cosmetic or pharmaceutical applications was described in US 4 026 818 but the formulation requires the presence of hydroxylic solvents and utilises a surfactant system which is unsuitable for shampoo applications. EP O 598 335 describes the use of various cubic phases including  $I_1$  phases as laundry prespotters and for other cleaning formulations. It does not suggest how such phases could be used to suspend oil or form microemulsions. Normally attempts to

suspend oil in surfactant mesophases result in coarse droplets of oil being suspended in the aqueous phase of a structured surfactant.

Our invention provides a concentrated personal cleansing composition comprising, by weight of the composition, at least 20% water, 10 to 40% total surfactant and 2 to 40% of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.

The surfactants are preferably selected to provide an  $I_1$  phase over a comparatively broad surfactant concentration range e.g. more than  $\pm 5\%$  or greater, which range typically lies above 15% by weight total surfactant based on the weight of the composition e.g. between 20% and 40% by weight surfactant usually between 25% and 60%.

The surfactants are preferably selected to provide an  $I_1$  phase which melts above 30°C e.g. above 35°C, most preferably above 40°C. Preferably the  $I_1$  phase melts at a temperature substantially below 100°C, e.g. below 90°C, more preferably below 80°C, most preferably below 70°C, especially below 60°C, typically below 55°C, usually below 50°C.

The surfactant mixture preferably has a mean HLB based on the molar proportions of the components between 10 and 15 e.g. 11 to 14. The surfactants preferably comprise non-ionic surfactants such as ethoxylated alcohols. It has been found that highly ethoxylated fatty alcohols, e.g. more than 10 EO groups, preferably more than 15 EO groups, especially 18 to 50 EO groups form  $I_1$  phases particularly readily.

Other non-ionic surfactants which may be present include:-

alkyl phenol ethoxylates, fatty acid ethoxylates, fatty acid monoalkylolamide ethoxylates, fatty alcohol propoxylates, fatty amine alkoxyates and fatty acid glyceryl ester ethoxylates. Other non-ionic compounds suitable for inclusion in compositions of the present invention include mixed ethylene oxide propylene oxide block copolymers, low relative molecular mass polyethylene glycols e.g. PEG600 and PEG200, ethylene glycol monoesters, amine oxides and alkyl polyglycosides, alkyl sugar esters including alkyl sucrose esters and alkyl oligosaccharide ester, alkyl capped polyvinyl alcohol and alkyl capped polyvinyl pyrrolidone.

Compositions of the invention may also comprise anionic surfactants, in addition to or instead of non-ionic surfactants. Anionic surfactant may comprise a C<sub>10-20</sub> alkyl benzene sulphonate or an alkyl ether sulphate which is preferably the product obtained by ethoxylating a natural fatty or synthetic C<sub>10-20</sub> e.g. a C<sub>12-14</sub> alcohol with from 1 to 20, preferably 2 to 10 e.g. 3 to 4 ethyleneoxy groups, optionally stripping any unreacted alcohol, reacting the ethoxylated product with a sulphating agent and neutralising the resulting alkyl ether sulphuric acid with a base. The term also includes alkyl glyceryl sulphates, and random or block copolymerised alkyl ethoxy/propoxy sulphates.

The anionic surfactant may also comprise, for example, C<sub>10-20</sub> e.g. C<sub>12-18</sub> alkyl sulphate.

The surfactant may comprise a C<sub>8-20</sub> e.g. C<sub>10-20</sub> aliphatic soap. The soap may be saturated or unsaturated, straight or branched chain.

Preferred examples include dodecanoates, myristates, stearates, oleates, linoleates, linoleates and palmitates and coconut and tallow soaps.

The surfactant may include other anionic surfactants, such as olefin sulphonates, paraffin sulphonates, taurides, isethionates, ether sulphonates, ether carboxylates, aliphatic ester sulphonates e.g. alkyl glyceryl sulphonates, sulphosuccinates or sulphosuccinamates.

The cation of any anionic surfactant is typically sodium but may alternatively be potassium, lithium, calcium, magnesium, ammonium, or an alkyl ammonium having up to 6 aliphatic carbon atoms including isopropyl ammonium, monoethanol ammonium, diethanol ammonium, and triethanol ammonium.

Ammonium and ethanol ammonium salts are generally more soluble than the sodium salts. Mixtures of the above cations may be used.

The composition may contain amphoteric surfactants such as betaines sulphobetaines, amido betaines or imidazoline betaines.

The I<sub>1</sub> phase may be conveniently prepared by mixing the oil and oil soluble surfactant and adding sufficient water to the water soluble surfactant to maintain a lamellar phase. The oil and oil soluble surfactant may be stirred into the lamellar composition at elevated temperature, above the melting point of the desired I<sub>1</sub> phase. The composition is then diluted with hot water until a microemulsion is formed and then cooled to solidify it into the I<sub>1</sub> phase.

The oil is preferably a mineral oil (e.g. a low molecular weight petroleum ether having, for example, a boiling point below 120°C e.g. below 100°C especially below 80°C) or a lower molecular weight fatty ester (e.g. one having less than 25 carbon atoms) such as isopropyl esters of lauric isostearic or palmitic acids or their ethyl analogues. Other oils, including higher mol weight fatty esters, e.g. oleyl oleate, fatty glycerides, terpene oils such as limonene or silicone oils may present difficulties in providing clear compositions. Such oils can nevertheless be incorporated in clear formulations by blending with sufficient mineral oil (preferably low molecular weight mineral oil). The amount required varies according to the nature of the oil. Typically the blend contains at least 16%, based on the total weight of oil, of the mineral oil, especially 30 to 80%, typically 40 to 60%. Particularly preferred are vegetable oils such as coconut, evening primrose, groundnut, meadow foam, apricot kernel, peach kernel, avocado, jojoba and olive oil.

Oil soluble cosmetic or topical pharmaceutical ingredients may be dissolved in the oil including antiseptics, styptics, antidandruff agents such as zinc omadine (zinc pyrithione) and selenium disulphide, proteins, emollients such as lanolin, isopropyl myristate, glyceryl isostearate or propylene glycol distearate, dyes, perfumes and waxes. Water insoluble particulate solids including exfoliants such as talc, clays, polymer beads, sawdust, silica, seeds, ground nutshells and dicalcium phosphate, pearlisers such as mica or glycerol or ethylene glycol mono- or di-stearate, glitter additives and sunscreens such as titanium dioxide may be dispersed in the hot microemulsion prior to cooling. Porous particles (so called micro-sponges) containing absorbed active ingredients or gelatin or other microcapsules may be suspended. Other active ingredients which may be suspended include insect repellants and topical pharmaceutical preparations, e.g. preparations for treatment of acne, fungicides for athlete's foot or ringworm or antiseptics or antihistamines. Pigments, such as the iron oxides, may also be added.

Electrolytes tend to break  $I_1$  phase structure and are preferably present in concentrations below 10% based on total weight of the compositions, more preferably below 5%, e.g. 0 to 3%, most preferably 0 to 1%. Generally we prefer that electrolyte be substantially absent. Adventitious chloride or sulphate present as impurities in the surfactant can be tolerated. Small amounts of builder such as citrates, pyrophosphates, polyphosphates may optionally be included.

Water soluble solvents are generally undesirable and are not required to form stable  $I_1$  structures according to the invention. We therefore prefer that they should be substantially absent. Although small amounts of, for example, ethanol or propanol or of a water miscible polyhydric alcohol or alcohol ester may sometimes be desired for special purposes, they are preferably present in amounts less than 5% by weight, more preferably less than 3% by weight, most preferably less than 2% by weight, e.g. less than 1% by weight.

The composition may optionally contain hydrotropes such as sodium lower alkyl benzene sulphonate e.g. sodium toluene, xylene or cumene sulphonate or urea, however these are not generally necessary and are not generally preferred. We prefer that these should be present in quantities less than 5% by weight, more preferably less than 4%, especially less than 2% e.g. 0 to 1%. They may be useful occasionally to avoid haziness of the gel.

The total amount of water is preferably from 25 to 60% by weight of the composition, more preferably 30 to 50%, e.g. 35 to 50%. The total weight percentage of surfactant based on the weight of the composition is preferably from 15 to 35%, e.g. 20 to 30%. The proportion of oil is preferably greater than 5%, more preferably greater than 8%, e.g. 10 to 30%, especially 15 to 25% by weight based on the weight of the composition. The oil soluble surfactant is preferably present in a proportion of more than 1:5 based on the weight of oil, more preferably from 1:2 to 5:1. The oil soluble surfactant preferably has an HLB of from 3 to 9 e.g. 4 to 8.

The weight ratio of water soluble surfactant to oil soluble surfactant is preferably 1:1 to 30:1, more preferably 2:1 to 20:1, typically 3:1 to 15:1, e.g. 4:1 to 10:1. The water soluble surfactant preferably has an HLB greater than 12, more preferably greater than 13, especially 14 to 19.

The product may be cast into shaped bodies or formed into particles or granules, e.g. by spray cooling a hot solution of the  $L_1$  phase formed on melting the composition.

The composition may be converted into a microemulsion phase by addition of water, by heating above the melting point or by adding electrolyte such as salt and the invention includes  $L_1$  phases when so prepared.

The invention will be illustrated by the following examples:

**Example 1**

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<u>Component</u>	<u>Solids (%)</u>	<u>w/w (%)</u>
MINERAL OIL (100%)	20	20
"EMPICOL"® 0251/70J (70%)	11.2	16
"EMPIGEN"® BB (30%)	4.8	16
"GLUCAPON"® 215 CS UP (65%)	6	9.2
"EMPILAN"® KB2 (100%)	7.5	7.5
SODIUM CHLORIDE (100%)	2	2
PERFUME (100%)	0.5	0.5
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.1	0.1
CITRIC ACID (100%)	0.2	0.2
BENZOIC ACID (100%)	0.3	0.3
SODIUM HYDROXIDE (47%)	0.1	0.2
WATER	---	Balance

The method of mixing comprised the following steps:-

1. Charge 50% of water
2. Heat to 60°C
3. Add EDTA, sodium benzoate, citric acid and 47% NaOH dissolve with stirring
4. Add "EMPIGEN" BB
5. Add mineral oil and disperse with stirring
6. Add "EMPILAN" KB 2 and mix thoroughly
7. Add "EMPICOL" 0251/70j
8. Add remaining water
9. Add "GLUCAPON" 215 CS UP
10. Add further KB 2 until clear
11. Cool
12. Add evaporated water
13. Adjust pH

#### **Physical Data**

pH (10%)	: 5.5 ± 0.1	Density @ 20°C	: 1.0 ± 0.1 g cm <sup>-3</sup>
Solids (%)	: ~ 53% (typical)	Appearance	: Clear or Hazy Gel
Odour	: Characteristic	Set Point (typical)	: 30°C
Viscosity @ 20°C : N/A			

The product was examined by x-ray diffraction and exhibited peaks at 13.145nm (intense and sharp), 7.943nm (ill defined) and 6.355nm (small), indicating cubic symmetry, and formed a clear microemulsion on dilution or heating. The latter gave good even distribution of oil applied to skin.

#### **Example 2**



The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<u>Component</u>	<u>Solids (%)</u>	<u>w/w (%)</u>
MINERAL OIL (100%)	15	15
"EMPICOL"® CDL30J/35 (22%)	8	35.4
"EMPIGEN"® BB (30%)	8	26.7
"EMPICOL"® 0785 (40%)	2	5
"EMPILAN"® KB2 (100%)	6	6
"EMPILAN"® KB6 (100%)	6	6
CITRIC ACID (100%)	0.5	0.5
PERFUME (100%)	0.2	0.2
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.2	0.2
"KATHON"®	---	0.2
WATER	---	Balance
<b>TOTAL</b>	<b>45.8</b>	<b>100</b>

#### Physical Data

Appearance	: Clear Liquid/Gel	Odour	: Characteristic Odour
Solids	: 36.5% (typical)	pH (100%)	: 5.5 - 6.5 (typical)
Odour	: Characteristic	Set Point	: 20 ± 5°C

Viscosity (Carrimed Rheometer @ 20°C : N/A

The product had small angle x-ray diffraction peaks characteristic of cubic symmetry and formed a clear microemulsion on dilution with water or warming. The latter gave good even deposition of oil on skin.

**Examples 3 and 4**

The following ingredients were mixed at 60°C and cooled to form ringing gels:

Component	1		2	
	Solids (%)	w/w (%)	Solids (%)	w/w (%)
"EMPIGEN"® CDL30J/35 (22%)	8	36.4	8	36.4
"EMPIGEN"® BB (30%)	8	26.7	8	26.7
"EMPICOL"® LB40 (40%)	4	7.5	3	7.5
"EMPICOL"® CVH (90%)	4	4	---	---
"EMPILAN"® KB2 (100%)	5.5	5.5	6	6
TRIETHANOLAMINE (100%)	1.1	1.1	---	---
CITRIC ACID	1	0.75	0.75	0.75
ETHYLENE DIAMINE				
TETRACETIC ACID	0.05	0.05	0.05	0.05
"KATHON"® CG (100%)	0.05	0.05	0.05	0.05
LIGHT MINERAL (100%)	14	14	20	20
WATER	---	Balance	---	Balance
<b>TOTAL</b>	<b>45.7</b>	<b>100</b>	<b>46.1</b>	<b>100</b>
<b>Appearance</b>	Clear Gel		Clear Gel	

The following ingredients were mixed at 60 °C and cooled to form a clear 'ringing' gel.

### **Example 5**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	18	18
"EMPICOL" ® 0251 70 J (70 %)	12	17.2
"EMPICOL" ® CED5 FL (100 %)	5	5
"EMPILAN" ® KBE2 (100 %)	3	3
"EMPILAN" ® KB6 (100 %)	3	3
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

### **Example 6**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
HEAVY MINERAL OIL ("KRISTOL" ® M70) (100 %)	18	18
"EMPICOL" ® 0251 70 J (70 %)	10.5	15
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" ® KB2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.5	1.0
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

**Physical Data**

Density @ 20 <sup>0</sup> C	: 1.0 +/- 0.1	pH (10 %)	: 5.5 +/- 0.5
Appearance: Clear or hazy gel		Odour	: Characteristic
Set point (typical): 35 +/- 5 <sup>0</sup> C		Viscosity @ 20 <sup>0</sup> C:	N/A

**Method for examples 5 and 6**

- i) Charge water and heat to 60<sup>0</sup>C.
- ii) Add EDTA, sodium benzoate, citric acid and NaOH. Dissolve with stirring.
- iii) Add "EMPICOL" CED5 FL and mix thoroughly.
- iv) Add glycerol.
- v) Add NaCl and disperse with stirring.
- vi) Add "EMPILAN" KBE2 and "EMPILAN" KB6 or "EMPILAN" KB12. Disperse with stirring.
- vii) Add "EMPIGEN" BB.
- viii) Add mineral and disperse with stirring.
- ix) Add "EMPICOL" 0251 70J and disperse with stirring.
- x) Add additional nonionic surfactant to clear (if necessary).
- xi) Cool to 40<sup>0</sup>C.
- xii) Add evaporated water
- xiii) Adjust pH and offload.

**Example 7**

<b>Component</b>	<b>Solids (%)</b>	<b>W/W (%)</b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	9	9
DOW CORNING DC 556 SILICONE FLUID (100 %)	9	9
"EMPICOL" 0251 70 J (70 %)	12	17.2
"EMPICOL" CED5 FL (100 %)	5	5
"EMPILAN" KB2 (100 %)	3.5	3.5
"EMPILAN" KB12 (100 %)	3.5	3.5
"EMPIGEN" BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

The formulation forms a microemulsion at 60<sup>0</sup>C and forms a gel when cooled to ambient temperature.

**Example 8**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
HEAVY MINERAL OIL ("KRISTOL" ® M70) (100 %)	15	15
"CERAPHYL" ® GA-D (100 %)	5	5
"EMPICOL" 0251 70 J (70 %)	12	17.2
"EMPICOL" CED5 FL (100 %)	5	5
"EMPILAN" KBE2 (100 %)	3.0	3.0
"EMPILAN" KB12 (100 %)	4.5	4.5
"EMPIGEN" BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

A hazy/opaque emulsion is formed at 60°C and cools to form a clear 'ringing' gel at ambient temperature.

**Physical Data**

Density @ 20°C : 1.0 +/- 0.1

pH (10 %) : 5.5 +/- 0.5

Appearance: Clear or hazy gel

Odour : Characteristic

Set point (typical): 35 +/- 5°C

Viscosity @ 20°C: N/A

**Method for examples 7 and 8**

- i) Charge water and heat to 60°C.
- ii) Add EDTA, sodium benzoate, citric acid and NaOH. Dissolve with stirring.
- iii) Add "EMPICOL" CED5 FL and mix thoroughly.
- iv) Add glycerol.
- v) Add NaCl and disperse with stirring.
- vi) Add "EMPILAN" KBE2 and "EMPILAN" KB12. Disperse with stirring.
- vii) Add "EMPIGEN" BB.
- viii) Blend 50/50 oil phase - oil and cosmetic ingredient. Add to aqueous surfactant solution. Disperse with stirring to form homogeneous emulsion.
- ix) Add "EMPICOL" 0251 70J and disperse.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Adjust pH and offload.

If gel is opaque, re-heat and add additional nonionic surfactant or water.

**Example 9**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	10	10
"MIGLYOL" ® 810/812S	10	10
"EMPICOL" ® 0251 70 J (70 %)	11	15.7
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" ® KBE2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	3.5	3.5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.5	1
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

Hazy emulsion clears to form a microemulsion on cooling and 'ringing' gel is obtained at ambient temperature.



**Example 10**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	10	10
"MIGLYOL" ® 818 (100 %)	10	10
"EMPICOL" ® 0251 70 J (70 %)	11	15.7
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" ® KBE2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.5	1
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

Forms a microemulsion at 60°C and a 'ringing' gel is obtained after cooling.

**Example 11**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	10	10
"MIGLYOL" ® 840	10	10
"EMPICOL" ® 0251 70 J (70 %)	11	15.7
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" KBE2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.5	1
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

**Physical Data**

Density @ 20°C	: 1.0 +/- 0.1	pH (10 %)	: 5.5 +/- 0.5
Appearance:	Clear or hazy gel	Odour	: Characteristic
Set point (typical):	35 +/- 5°C	Viscosity @ 20°C:	N/A

**Method for examples 9, 10 and 11**

- i) Blend 50/50 oil phase – oil and cosmetic ingredient. Heat to 60°C.
- ii) Add glycerol and stir to disperse.
- iii) Add “EMPILAN” KBE2 and “EMPILAN” KB12. Disperse with stirring.
- iv) Add “EMPICOL” CED5 FL.
- v) Add “EMPIGEN” BB.
- vi) Add “EMPICOL” 0251 70J.
- vii) Add EDTA, citric acid, sodium benzoate and NaCl. Disperse with stirring.
- viii) Add water.
- ix) Add NaOH.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Adjust pH and offload.

**Example 12**

<b>Component</b>	<b>Solids (%)</b>	<b>W/W (%)</b>
EMOLLIENT – FATTY ACID ESTER (100 %)	20	20
“EMPICOL” ® 0251 70 J (70 %)	12	17.2
“EMPICOL” ® CED5 FL (100 %)	5	5
“EMPILAN” ® KB6 (100 %)	5	5
“EMPIGEN” ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

Clear gels have been prepared using the following fatty acid esters:

Isopropyl laurate (“ESTOL” ® IPL 1505)  
 Isopropyl myristate (“ESTOL” ® IPM 1512)  
 Isopropyl palmitate (“ESTOL” ® IPP 1517)  
 Isopropyl isostearate (“SCHERCOMOL” ® 318)

**Physical Data**

Density @ 20°C : 1.0 +/- 0.1

pH (10 %) : 5.5 +/- 0.5

Appearance: Clear or hazy gel

Odour : Characteristic

Set point (typical): 35 +/- 5°C

Viscosity @ 20°C: N/A

**Method for example 12**

- i) Heat oil phase to 60°C.
- ii) Add “EMPILAN” KB6 and stir to disperse.
- iii) Add glycerol and stir to disperse.
- iv) Add “EMPIGEN” BB.
- v) Add “EMPICOL” CED5 FL.

- vi) Add "EMPICOL" 0251 70J.
- vii) Add EDTA, NaCl, sodium benzoate and citric acid. Stir to disperse.
- viii) Add water.
- ix) Add NaOH.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Check pH (10 %).
- xiii) Adjust pH and offload.

The products in each case exhibited cubic symmetry and formed clear microemulsions or dilution with water or heating. The registered trade marks noted above have the following significance:-

"EMPICOL" CVH is a C<sub>8</sub> alkyl ether carboxylic acid

"EMPICOL" LB40 is a C<sub>8</sub> C<sub>10</sub> alkyl sulphate

"EMPICOL" 0251/70J is a C<sub>12-14</sub> alkyl 3 mole ethoxy sulphate

"EMPICOL" 9758 is a C<sub>10</sub> alkyl sulphate

"EMPICOL" CED 5FL is lauryl 6 mole ethoxy carboxylic acid

"EMPIGEN" BB is a C<sub>12-14</sub> alkyl betaine

"EMPIGEN" CDL is coconut amphi acetate

"EMPILAN" KB2 is a C<sub>12-14</sub> alkyl 2 mole ethoxylate

"EMPILAN" KB6 is a C<sub>12-14</sub> alkyl 6 mole ethoxylate

"EMPILAN" KB12 is a C<sub>12-14</sub> alkyl 12 mole ethoxylate

"GLUCAPON" 215CS is a C<sub>8-10</sub> alkyl polyclucoside D.P. 1.5

"KATHON" CG is a proprietary biocide

"DOW CORNING" DC556 is phenyl trimethicone

"CERAPHYL" GA-D is maleated soya bean oil

"MIGLYOL" 810/812S is capric/caprylic triglyceride

"MIGLYOL" is capric/caprylic/linoleic triglyceride

"MIGLYOL" 840 is dipropylene glycol dicaprylate/dicaprate

**CLAIMS**

1. A concentrated personal cleansing composition comprising, by weight of the composition, at least 20% water, 10 to 40% total surfactant and 2 to 40% of oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.
2. A composition according to claim 1 wherein the total surfactant has a mean HLB between 10 and 15.
3. A composition according to claim 1 wherein said oil comprises a mineral, fatty ester, glyceride, terpene or silicone oil
4. A composition according to either of claims 1 and 3 wherein the oil comprises at least 16% based on the weight of oil, of a mineral oil.
5. A method for preparing a composition according to claim 1 comprising : (i) forming a mixture (a) of said oil and said oil soluble surfactant; (ii) mixing said mixture (a) with a mixture (b) of said water soluble surfactant and sufficient water to form a lamellar phase with said water soluble surfactant; (iii) maintaining said mixture of (a) and (b) above the  $I_1/L_1$  transition temperature of said composition while diluting said mixture of (a) and (b) with water to form said composition; and (iv) cooling said composition below the  $I_1/L_1$  transition temperature.

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ance Notes on Codes and Abbreviations" appearing at the begin-  
ning of each regular issue of the PCT Gazette.

(54) Title: PERSONAL CARE FORMULATIONS

(57) Abstract: Personal care compositions contain at least 20 % water, 10 to 40 % total surfactant and 2 to 40 % of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (a) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (b) a hydrophilic surfactant having an HLB greater than 11 in a weight proportion of from 1:1 to 1:30 based on the weight of (a), said water surfactant and oil being present in proportions adapted to form an I<sub>1</sub> phase having an I<sub>1</sub>/L<sub>1</sub> transition temperature greater than 25 °C.

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# INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 00/05341

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61K7/00 A61K7/06 A61K7/50 A61K7/48 C11D17/00  
C11D1/94 C11D3/18

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, CHEM ABS Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 14 67 825 A (CHESEBROUGH_PONDS INC.) 2 January 1969 (1969-01-02)	1-5
Y	page 1, paragraph 1 - paragraph 2 page 2, paragraph 3 page 3, paragraph 3 page 4, paragraph 4 -page 5, paragraph 1 page 9, paragraph 4 -page 11, paragraph 2 examples claims	1-5
A	US 4 772 427 A (DAWSON ANDREW F ET AL) 20 September 1988 (1988-09-20) abstract column 3, line 21 - line 24 column 3, line 41 -column 4, line 5 column 4, line 22 - line 34 examples 1,3,4,7 claims	1-3



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

### \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
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- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*8\* document member of the same patent family

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# INTERNATIONAL SEARCH REPORT

International Application No

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>US 5 756 108 A (BIATRY BRUNO ET AL)  26 May 1998 (1998-05-26)  abstract  column 1, line 1 -column 2, line 16  column 2, line 63 -column 5, line 7  column 7, line 32 -column 8, line 4  examples 1,2,6  claims</p> <p style="text-align: center;">---</p>	1-5
A	<p>US 5 474 776 A (KOYANAGI HIDENOBU ET AL)  12 December 1995 (1995-12-12)  abstract  column 2, line 4 - line 19  column 2, line 35 -column 5, line 30  table 3  examples 3,4  claims 1,2</p> <p style="text-align: center;">---</p>	1-3
A	<p>PATENT ABSTRACTS OF JAPAN  vol. 1999, no. 06,  31 March 1999 (1999-03-31)  &amp; JP 08 143420 A (PROCTER &amp; GAMBLE  CO:THE), 4 June 1996 (1996-06-04)  abstract</p> <p style="text-align: center;">---</p>	
A	<p>EP 0 490 053 A (WELLA AG)  17 June 1992 (1992-06-17)  abstract  page 2, line 35 -page 3, line 56</p> <p style="text-align: center;">---</p>	1-3
P,Y	<p>RODRIGUEZ, CARLOS ET AL: "Cubic phase  -based concentrated emulsions"  J. COLLOID INTERFACE SCI. (2000), 223(2),  197-204,  XP000979610  abstract  page 197, column 1, paragraph 2 -column 2,  paragraph 4  figure 1  page 198, column 1, paragraph 5  page 199, column 1, paragraph 2 -page 200,  column 1, paragraph 2  page 202, column 1, paragraph 1 -page 203,  column 1, paragraph 1</p> <p style="text-align: center;">-----</p>	1-5



**FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210**

## Continuation of Box I.2

Present claims 1-5 relate to a composition and a method defined by reference to the parameter "an I1 phase having an I1/L1 transition temperature greater than 25 degrees C". The use of this parameter in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameter the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Consequently, the search has been restricted to compositions having the properties of an I1 phase as described in the description (p. 1, par. 5, p. 5, par. 3 and 4, and p. 6, par. 2), namely compositions in the form of clear gels, ringing gels or having a visous isotropic or a "VI" phase or a cubic liquid crystalline phase, or being immobile, non-Newtonian, optically isotropic and transparent.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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